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RETARDATION OF GELATION IN HIGH-TEMPERATURE–SHORT-TIME STERILE MILK CONCENTRATES WITH POLYPHOSPHATES

SUMMARY

Pilot plant and commercial plant scale experiments confirmed small-scale laboratory findings on the ability of polyphosphates to stabilize markedly high-temperature–short-time (HTST) sterile milk concentrates, both against heat coagulation during sterilization and gelation during storage. Thus, the storage life, 45 days at 70 F, of three-to-one sterile concentrates was prolonged to more than 441 days when 0.4 lb of a commercial polyphosphate with an average of 4.8 phosphorus atoms per chain was added per 100 lb milk solids in the sequence of operations—forewarming, concentration, addition of additive, sterilization, and homogenization. In the sequence of operations—forewarming, homogenization, addition of additive, sterilization, and concentration, the storage life, seven days, of control concentrates was extended to 240 days when polyphosphate was added. Because of the instability toward heat of three-to-one concentrates, the presence of additive during sterilization and superior homogenization following sterilization are necessary conditions for the prevention of undue phase separation (creaming and sediment formation) during prolonged storage. True ultra-high-short processing of sterile milk concentrates is best achieved in a sequence of operations in which sterilization precedes concentration, for then the defects of creaming and sediment formation are avoided. Thermal coefficients of the rate constants governing color development, sediment formation, flavor development, thickening, and gelation appear to be of such a character that the practice of storing at temperatures of approximately 70 F and lower has much to commend it.

Of four classes of additives which in laboratory scale experiments (5) effectively retarded age thickening of HTST sterilized milk concentrates, the polyphosphates—the most promising—were selected for studies on a larger scale. The results of pilot plant studies confirmed laboratory findings. These will be presented and discussed.

EQUIPMENT, APPARATUS, AND MATERIALS

The plant used in this study comprised the following units: Two types of evaporators—a Rogers[1] vacuum pan and a Wiegand-Harris[1] tubular falling-film evaporator; a Mallory[1] type sterilizer consisting of coiled-tube heating, holding and cooling sections (see Table 1), with an in-line two-stage homogenizer valve at the end of the cooling section; a small vacuum pan for aseptic evaporation with condenser and distillate receiver; an homogenizer pump for forcing the milk through the sequence of heat exchangers and holding tube; a high-pressure homogenizer for homogenizing before or after sterilization at pressures as high as 8,000 psi; a 20-liter pyrex glass reservoir for collecting sterile concentrate; a filling pipette of approximately 150-ml capacity; a specially designed steam tunnel (3) in which presterilized cans in their passage were flushed with steam, then inverted, filled, and covered aseptically; a can seamer with provisions for seaming under vacuum or in an inert atmosphere.

The apparatus for laboratory processing and study has been described in previous publications (4).

Milk was obtained from the Beltsville, Maryland, herd of the U. S. Department of Agriculture. The polyphosphate employed in our studies was an amorphous material known as Quadrafos, with an average of 4.8 phosphorus atoms per chain. It was obtained from the Rumford Chemical Company,[1] Rumford, Rhode Island.

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[1] The use of trade names is for the purpose of identification only, and does not imply endorsement of the product or its manufacturer by the U. S. Department of Agriculture.

TABLE 1

Data for Mallory heating, holding, and cooling system

Section	Tube inside diameter	Coil diameter	Flow rate per minute	Velocity	Reynolds no.[a]	Transit time 280(295°)	Fo at 280 F[d]	Fo at 295 F[e]
	(inches)	*(inches)*	*(cu in)*	*(ft per sec)*		*(sec)*	*(min)*	*(min)*
Heating	0.19	12.1	6.6	19.3	27,900	2.2(2.2)	0.1	0.6
Holding	0.31	12.1	6.6	7.2	17,000	14.5(0.6)	9.8[b]	3.2[c]
Cooling	0.31	10.0	6.6	7.2	17,000	9.3(9.3)	0.0	0.0

[a] For straight tubing and liquid with viscosity equal to 0.01 poise, and density equal to 1.000.

[b] Allowance made for 2 F temperature drop. Average temperature—279 F.

[c] For holding tube 51 in. in length.

[d] Time required to achieve the same lethal effect at 250 degrees as that achieved with indicated heat treatment, and steam chest temp—324 F, and initial temp—84 F.

[e] Steam chest temperature—343 F.

Four constant-temperature rooms set at 40, 70, 86, and 99 F were available for storage of samples in 5.5-oz cans (2½ by 2½ in.).

Measurements. Viscosity measurements at 30 C were made with a transpiration type capillary viscometer (2), operating under a maximum shearing stress of about 175 dynes per square centimeter. Plasticity measurements were made by varying the applied pressure, measuring the volume displacement at a number of pressures, and plotting the average shearing stress (corrected for the variable back-head of fluid in the viscometer) as abscissae, and the mean rate of shear as ordinates. All concentrates tested exhibited non-Newtonian behavior. Increases in body during storage usually reflected increases in both yield value and consistency. Inasmuch as plasticity measurements were time-consuming and furnished more information than was really necessary, viscosity measurements at one definite applied stress per se were largely relied upon for following body changes.

Sediment formation was observed periodically in cans emptied at an angle of 45 degrees during a 1-min interval. The percentage sediment by weight was determined from the weight of the concentrate and the weight of sediment, and the thickness from the weight of sediment and its density (assumed to be the same as the concentrate).

This technique, although largely applicable, failed when the sediment was loose or when the surrounding concentrate was extremely heavy-bodied. Sediment was redispersed by placing it in a fine wire-mesh stemless funnel, the apex of which was located beneath the surface of the sediment-free concentrate. Wet grinding and filtering the sediment through the screen in the presence of a small volume of continuously renewable concentrate, permitted uniform and homogeneous dispersion of the sediment in a reproducible manner prior to a viscosity measurement.

Color indices were obtained in the manner described by Webb and Holm (8). Fat and total solids were determined according to the Mojonnier procedure (6).

PROCEDURE

HTST processing allows for a wide latitude in varying the order of processing steps. The sequences listed below were followed in our studies:

1. Homogenization, forewarming, concentration, addition of additive, sterilization—the H-F-C-A-S sequence.
2. Forewarming, concentration, addition of additive, sterilization, homogenization—the F-C-A-S-H sequence.
3. Forewarming, concentration, sterilization, homogenization, the addition of additive—the F-C-S-H-A sequence.
4. Forewarming, homogenization, addition of additive, sterilization, concentration—the F-H-A-S-C sequence.

Only Items 2 and 4 were employed to any great extent.

In the processing according to the F-C-A-S-H sequence, milk was first standardized to contain fat and solids in the ratio 1:3.3. The standardized milk was forewarmed usually at 280 F for 15 sec, cooled to about 120 F, and pumped either into the Rogers vacuum pan or the evaporating section of the Wiegand falling film evaporator. To the concentrate either water or additive solution was added to bring the solids or solids and additives to the desired levels. The concentrate was forced through the heating section of the Mallory, and kept at the holding temperature as it passed through the holding section, from whence the concentrate was cooled to homogenization temperature in the cooling section prior to its passage through either an in-line homogenizer valve or a high-pressure homogenizer. Continuing in its motion, the sterile homogenized concentrate passed through a cooler before its introduction into the receiver. The manner of processing according to other sequences may be readily inferred.

RESULTS AND DISCUSSION

Effect of additives in the F-C-A-S-H sequence. The F-C-A-S-H sequence appears to be the preferred one at the present time. Standardized forewarmed milk was concentrated batchwise in the Rogers pan. The concentrates following sterilization but before homogenization showed evidence of heat damage (graininess), the extent of which was much more pronounced in the control than in either of the additive-containing concentrates, and practically unobjectionable in the polyphosphate-containing milk. Homogenization, although it erased differences in appearance between concentrates, did not eliminate differences in body as measured by viscosity. Decreasing the temperature of storage from 99 to 70 F brought with it an increase in storage life to an extent which was much more pronounced in the polyphosphate-, *P,* containing samples than in either the control, *C,* or the monophosphates-, *M,* containing samples (see Table 2). The gain in stability as effected by the addition of polyphosphate was particularly striking in samples stored at 70 F for, whereas the storage life of the *M*-containing concentrate was only 16 days and that of the control, 45 days, the storage life of the *P*-containing concentrate had not ended even after 441 days. Considering only the *P*-containing concentrates for the moment, the gain with respect both to the rate of sediment formation and color change, as these were influenced by a decrease in temperature, was no less striking than that achieved with respect to age-thickening and gelation.

The dual function of the polyphosphate is amply demonstrated by the experiments, substantiating in this respect data of laboratory experiments (5), for the additive not only stabilizes sterile milk concentrates against gelation during storage, but it also stabilizes against heat coagulation during HTST sterilization.

The data have a particular value in demonstrating that it is possible under optimum or near-optimum conditions to produce a highly body-stable HTST sterilized 3-to-1 concentrate. It remains only to determine in a general way what these optimum conditions are.

Influence of forewarming treatments. The data in Tables 3 and 4 illustrate the effect of

TABLE 2

Effect of added monophosphate buffer salts and a polyphosphate glass on the thickening during storage of HTST sterilized milk concentrates (36% solids). Concentrates processed according to the F-C-A-S-H sequence

			Storage time					
			180 Days			441 Days		
Additive [a]	Storage temp	Storage life	Viscosity	Sediment	Color index	Viscosity	Sediment	Color index [b]
	(F)	*(days)*	*(centipoises)*	[% *(inches)*]		*(centipoises)*	[% *(inches)*]	
Control (C)		45	Loose gel	....	..	......	....	......
Monophosphate (M)	70	16	Gel	....	..	......	....	......
Polyphosphate (P)		441	17.7	1.5(0.03)	1	25.8	1.5(0.03)	1–2
C		30	Gel	....	..	......	....	......
M	86	13	Gel	....	..	......	....	......
P		220	23.8	2.2(0.04)	2	52.3	10(0.19)	5
C		26	Gel	....	..	......	....	......
M	99	10	Gel	....	..	......	....	......
P		84	79.0	7.5(0.14)	8	......	....	......

[a] 0.4 lb of either a mixture of mono-, and disodium phosphate, or a commercial polyphosphate with an average of 4.8 phosphorus atoms per chain, added per 100 lb milk solids.

All milks forewarmed and sterilized at 280 F for 15 sec.

[b] Color scale ranges from one for unsterilized evaporated milk to 12. Average evaporated milk has color index of four or five. See Webb and Holm (8).

Viscosities of sterile concentrates—*C, M, P* were 53.0, 29.3, and 13.7 centipoises, respectively, before homogenization at 7,500-500 psi and 160 F.

TABLE 3

Influence of two forewarming treatments on storage qualities of HTST sterilized milk concentrates (37% solids) both in presence and absence of added polyphosphates. Concentrates processed according to the F-C-A-S-H sequence

Additive per 100 lb milk solids	Forewarming temp	Viscosity [c] before storage	Properties after 102 days storage at 40 F		Properties after 358 days at 40 F		Properties after 102 days at 70 F		Properties after 358 days at 70 F	
			Viscosity	Sediment	Viscosity	Sediment	Viscosity	Sediment	Viscosity	Sediment
(lb)	*(F)*	*(centipoises)*	*(Cp)*	[*%(inches)*]	*(Cp)*	[*%(inches)*]	*(Cp)*	[*%(inches)*]	*(Cp)*	[*%(inches)*]
0	280 [a]	32.6	49.8	1.5(0.03)	268 [d]	Loose gel	198 [d]	Loose gel	Gel	Gel
0	295 [b]	12.8	18.9	5.0(0.1)	43.7 [d]	Loose gel	32.5 [d]	Loose gel	Gel	Gel
0.6	280	15.1	20.9	1.5(0.03)	20.6	4.5(0.09)	15.4	4.5(0.09)	18.4	8(0.16)
0.6	295	11.6	17.7	2.0(0.04)	18.9	6.0(0.12)	15.4	6.0(0.12)	21.2	13(0.25)

[a] Forewarming time—15 sec; bringing-up time—2.2 sec; cooling time—9.3 sec.
[b] Forewarming time—0.6 sec; bringing-up time—2.2 sec; cooling time—9.3 sec.
[c] Max. shearing stress—175 dynes/cm^2; temp of measurement—30 C.
[d] Stirred-out viscosity. Sample stirred gently with spoon.

Concentrates homogenized at 7,500 psi after sterilization. Sterilization at 280 F for 15 sec. Sediments forming in additive-containing concentrates are dispersable. Additive, a polyphosphate glass containing an average of 4.8 phosphorus atoms per chain.
Color indices after 102 and 358 days at 40 degrees were 0-1, and 1, respectively.
Color indices after 102 and 358 days at 70 degrees were 0-1, and 2, respectively.

TABLE 4

Influence of several ultra-high-short processing schedules on storage qualities of milk concentrates (37% solids) in presence and absence of additive. Concentrates processed according to the F-C-A-S-H sequence

Additive per 100 lb milk solids	Forewarming temp	Storage life		Properties after 88 days at 40 F		Properties after 347 days at 40 F		Properties after 88 days at 70 F		Properties after 347 days at 70 F	
		70 F	40 F	Viscosity	Sediment	Viscosity	Sediment	Viscosity	Sediment	Viscosity	Sediment
(lb)	*(F)*	*(days)*	*(days)*	*(Cp)* [c]	[*%(inches)*]	*(Cp)*	[*%(inches)*]	*(Cp)*	[*%(inches)*]	*(Cp)*	[*%(inches)*]
0	295 [a]	50	80		1.5(0.03)	61.0 [d]	Loose gel	50.3 [d]	Lumpy	Gel	Gel
0	162 [b]	40	80	18.5	5.5(0.11)		Loose gel	51.5 [d]	Very lumpy	Gel	Gel
0.6	295	>347	>347	16.7	1.5(0.03)	17.9	2.0(0.04)	14.9	4.0(0.08)	14.4	16.0(0.32)
0.6	162	>347	>347	16.0	3.5(0.07)	17.7	6.0(0.12)	15.0	11.5(0.22)	16.0	22.0(0.44)

[a] Forewarming time—0.6 sec; bringing-up time—2.2 sec; cooling time—9.3 sec.
[b] Forewarming time—15 sec; bringing-up time—2.2 sec; cooling time—9.3 sec.
[c] Max. shearing stress—175 dynes/cm^2; temperature, 30 C.
[d] Stirred-out viscosity.

Concentrates homogenized at 7,500-500 psi and 160 F after sterilization at 295 F for 0.6 sec. Sediments forming in additive-containing concentrate were dispersable. Additive is a polyphosphate glass with an average of 4.8 phosphorus atoms per chain. Viscosities before storage were 8.9, 9.6, 10.8, and 11.3 centipoises for the concentrates as listed above.

a number of forewarming treatments on high-short and ultra-high-short sterilized concentrates, both in the presence and absence of polyphosphate. Forewarming at 280 F for 15 sec, compared with forewarming at 295 F for 0.6 sec, favored the formation during storage of a smaller amount of sediment in HTST-sterilized concentrates. Forewarming at 295 F for 0.6 sec, compared with forewarming at 162 F for 15 sec, was conducive to the formation during storage of a much smaller amount of sediment in ultra-high-short sterilized concentrates. No appreciable difference was observed between the amounts of sediment forming in high-short and ultra-high-short sterilized concentrates which had been forewarmed at 295 F for 0.6 sec.

However, under the experimental conditions the *Fo* values were not equal (see Table 1) and, hence, it is reasonable to expect that under conditions of constant *Fo*, more sediment would form in ultra-high-short sterilized concentrates.

Sediment, rather than gel formation, was the chief defect in additive-containing concentrates processed under high-short conditions without adequate forewarming. Sediment packed more closely in the polyphosphate-containing sample than in the control.

Sediment formation can assume serious proportions in HTST-processed concentrates. Much potential sediment arises during the sterilization step in view of the inherent instability of three-to-one whole milk concentrates. Thus, variations in the forewarming regime may be expected to bring about variation in heat stability and, consequently, in the degree of sedimentation. In this respect, and in addition to the results given above, forewarming at 280 F for 15 sec was found superior to forewarming at 195 F for 10 and 20 min, and to forewarming at 260 F for 42 sec.

Seehafer and Swanson (7), by means of solubility index determinations, and Wilson and Herreid (9) and Wilson et al. (10), in connection with sedimentation coefficient measurements and microscopic observations, have observed changes in aggregate formation and particle size brought about by various preheat and heat treatments which are in agreement with the observations reported in this paper.

Storage qualities of 2:1 sterile concentrates. Table 5 shows the results of an experiment in which the processing of 26% concentrates was effected in the presence and absence of additives under two processing sequences. Because of the exceptional thermal stability of the milk, it was possible to homogenize before sterilization without the development during sterilization of microscopically visible particles in the additive-containing sample.

TABLE 5

Storage qualities of heat-stable HTST sterilized 26% concentrates with and without additive; the influence of homogenization before sterilization. Concentrates processed according to the F-C-A-S-H and F-H-C-A-S-H sequence

Additive [a]	Storage temp	Storage life	Properties after 190 days storage		Properties after 308 days storage		Color index
			Viscosity at 30 C	Sediment	Viscosity at 30 C	Sediment	
	(F)	*(days)*	*(centipoises)*	*[%(inches)]*	*(centipoises)*	*[%(inches)]*	
Control (C1)	2	190	10.0	0	10.9	Crystalline deposit	0–1
Polyphosphate (P1)	40	>308	5.1	0	5.2	0	0–1
Control (C2)		165	12.6	0	Loose gel	Loose gel	0–1
Polyphosphate (P2)		>308	5.4	0	5.5	0	0–1
C1		140	15.1	Lumpy	Loose gel	Loose gel	1
P1	70	>308	4.2	0	4.4	0 [b]	1
C2		130	24.8	Lumpy	Loose gel	Loose gel	1
P2		>308	4.8	0	4.9	0 [b]	1
C1		114	Loose gel	Loose gel	Gel	Gel	2
P1	86	>308	4.7	1.0(0.02)	4.5	4.0(0.08)	2
C2		110	Loose gel	Loose gel	Gel	Gel	2
P2		>308	4.7	0	4.8	0 [b]	2

[a] C1 and P1 were homogenized at 7,500-500 psi and 160 F after sterilization. C2 and P2 were homogenized at 4,500-500 psi and 160 F after forewarming and at 7,500-500 psi and 160 F after sterilization. P1 and P2 contain 0.4 lb additive per 100 lb milk solids. The viscosities before storage of C1, P1, C2, and P2 were, respectively, 4.5, 4.1, 4.5, and 6.1 centipoises. The viscosity of P2 before rehomogenization was 4.5 centipoises.

[b] Thin layer of crystals present which analyzes as dicalcium phosphate dihydrate. Personal communication, R. A. Barackman, Victor Chemical Works, Chicago Heights, Illinois.

Processing according to the F-C-A-S-H and according to the F-H-C-A-S-H schedules were compared. The exceptional storage stability of the additive-containing concentrate, as well as the gain in stability in the presence of additive, were striking even at the highest temperature of storage, 86 F (see Table 5). With respect to gelation, one may, by extrapolation, reasonably expect a storage life approaching 2 yr at 86 F and corresponding greater storage stability at the lower storage temperatures. With respect to sediment formation, which was not serious under any circumstances, a small advantage accrued to the employment of the F-H-C-A-S-H sequence of steps. Color stability was excellent, and at the end of 308 days of storage at 86 F, the color index, two, was appreciably less than the index, five, associated with conventionally prepared evaporated milk. Visible cream layer and plug formation (observation not recorded in the table) were strikingly absent.

Results pertaining to storage lives and the differences in behavior between the controls and additive-containing concentrates paralleled the results obtained in laboratory micro-scale experiments in which the H-F-C-A-S sequence is normally followed (data not recorded).

Processing according to the F-H-A-S-C schedule. An inviting sequence of steps in HTST processing is one in which sterilization precedes rather than follows concentration; for, in such a sequence, problems associated with the processing of heat-unstable milks vanish. Table 6 shows the results of an experiment in which the sequence of steps, forewarming at 280 degrees for 15 sec, homogenization at 7,500-500 psi and 160 degrees, addition of additive, sterilization at 280 degrees for 15 sec and concentration were employed, both in the presence and absence of mono- and polyphosphates. What is perhaps the most striking feature in the data is the extreme lability shown by monophosphate-containing concentrates which form gels within a few days even at temperatures as low as 70 F. The control concentrates were hardly more stable, whereas the gain in stability brought about by the presence of polyphosphate was phenomenal. Thus, after 345 days of storage at 70 F, the polyphosphate-containing concentrate was still fluid although heavy-bodied, whereas the control concentrate had gelled within 12 days and the monophosphate-containing concentrate within six days. No less astonishing was the complete absence of sediment and creaming in the concentrates.

TABLE 6

Effect of added polyphosphate glass on storage life of three-to-one concentrates concentrated after sterilization at 280 F for 15 sec

Additive [a]	Storage temp	Storage life [b]	Storage time (days) 150: Viscosity	150: Sediment	345: Viscosity	345: Sediment	Remarks
	(F)	*(days)*	*(centipoises)*	*(per cent)*	*(centipoises)*	*(per cent)*	
Control (C)		24	Firm gel	....	Firm gel	....	Gel on 24th day
Monophosphate (M)	40	24	Firm gel	....	Firm gel	....	Gel on 24th day
Polyphosphate (P)		345	27.4	0	31.2	0	
C		7	Firm gel	....	Firm gel	....	Gel on 12th day
M	70	3	Firm gel	....	Firm gel	....	Gel on 6th day
P		240	27.8	0	56.0	0 [c]	
C		5	Firm gel	....	Firm gel	....	Gel on 12th day
M	86	3	Firm gel	....	Firm gel	....	Gel on 6th day
P		76	Gel	....	Gel	....	
C		6	Firm gel	....	Firm gel	....	Gel on 6th day
M	99	3	Firm gel	....	Firm gel	....	Gel on 3rd day
P		36	Gel	....	Firm gel	....	Loose gel on 75th day
		Solids *(per cent)*	Viscosity before storage *(centipoises)*				
C		37.5	30.6				
M		36.2	15.9				
P		36.4	15.0				

[a] Additive concn.—0.4 lb per 100 lb milk solids of polyphosphate glass containing an average of 4.8 phosphorus atoms per chain. Additive added after milk was forewarmed 15 sec at 280 degrees, and homogenized at 7,500-500 psi and 160 F.

[b] Time required for viscosity to reach a value equal to twice minimum.

[c] Thin layer of crystals present which analyzes as dicalcium phosphate dihydrate. Personal communication, R. A. Barackman, Victor Chemical Works, Chicago Heights, Illinois.

The polyphosphate-containing concentrate after 345 days storage at 70 degrees poured cleanly, to leave behind not a trace of protein residue in the can.

Processing according to the F-H-A-S-C sequence necessitates the troublesome operation of aseptic evaporation in vacuo, yet the arrangement of steps in which concentration follows sterilization is the only one which independently of the heat stability of the milk avoids coagulation during sterilization and its concomitant sediment formation during storage. It is the only arrangement consistent with true ultra-high-short processing for, otherwise, the forewarming step is quite necessary.

Well-defined gel characteristics are developed only when sterilization is unattended by coagulation, for then the elements of structure formation contained in the original milk persist; whereas, if sterilization follows concentration, the elements are inactivated to a greater or lesser degree by aggregate formation. Hence, an inverse relationship often prevails between sediment formation and gelation, an observation in accord with that of Seehafer and Swanson (7). Wilson et al. (10), as the result of sedimentation rate and microscopic studies, have observed that a substantial increase in particle size occurs in concentrated but not in unconcentrated milks as a consequence of ultra-high-short processing.

The structure formation in the absence of polyphosphate proceeds at a fantastically rapid rate. The effect of polyphosphate is indeed remarkable, for storage life at 70 F is increased from a matter of days to more than eight months. With the use of higher concentrations of additive, and with the employment of other types of polyphosphates, one may reasonably anticipate in view of the results of micro-scale experiments a further extension of storage life.

The question may be raised whether the differences in storage stability reflect the difference in the degree of aggregation of micelles as it is effected during sterilization. In view of the close agreement between the viscosities as well as the solubility indices of the sterile fluid milks (data not recorded), the experiment employing the concentration-following-sterilization sequence may be considered to indicate that differences in storage stability are not necessarily associated with differences in the degree of aggregation.

Results of plant-scale experiments. Results obtained in the pilot plant were sufficiently interesting to encourage experimentation on a commercial scale. Unfortunately, the specifications found advantageous in pilot plant studies required modification and relaxation to conform to the commercial facilities available (see Table 7). Thus, forewarming, which

TABLE 7

Processing data pertaining to plant run

		Heating section[a]				Holding[b]		Cooling	
Milk solids concentration	Additive concentration	Exit temp Heater 1	Exit temp Heater 2	Exit temp Heater 3	F. Heating	Exit temp Holding coil	F. holding	Homo. temp[c]	Filling temp[d]
(per cent)	*(lb per 100 lb solids)*	*(F)*	*(F)*	*(F)*	*(minutes at 250 F)*	*(F)*	*(minutes at 250 F)*	*(F)*	*(F)*
26	0	220	281		1.1	280	12.4	158	58
26	0.4	220	282		1.4	281	13.5	161	53
26	0.6	220	282		1.4	281	13.5	160	52
26	0.8	220	282		1.4	281	13.5	159	52
36	0	210	232	280	1.3	279	12.3	160	69
36	0.4	210	250	280	1.8	279	12.3	168	65
36	0.6	210	255	280	2.0	279	12.3	164	62
36	0.8	210	248	280	1.7	279	12.3	161	62

[a] Heating section consisted of 2 to 3 coils $\frac{7}{16}$-in. diameter mounted in 5-ft tower.

[b] Holding section consisted of two $\frac{7}{16}$-in. diameter coils for 2:1 concentrates and one $\frac{7}{16}$-in. diameter coil and one $\frac{1}{2}$-in. coil for 3:1. Heating time for 3:1 concentrates—22.5 sec, holding time—17 sec. Heating and holding time for 2:1 concentrates—15 sec.

[c] Homogenization temperature for 2:1 concentrates reached in 17.3 sec.

[d] Dole aseptic system employed.

Milks forewarmed at 195 F for 20 min. Concentrates homogenized at 2,000 psi after concentration and sterilization.

in our pilot plant was usually carried out at 280 F for 15 sec, was effected under the commercial plant schedule at 195 F for 20 min. The bringing-up time, 2.2 sec in pilot plant operations, was lengthened to 15-22.5 sec and thus contributed significantly to the Fo-value. Homogenization, usually effected at 7,500-500 psi in the pilot plant, was restricted to 2,000 psi in the plant. The equivalent sterilization time at 250 F (Fo, 9.9 sec) in pilot plant operations was lengthened to 14-15 sec.

These differences in processing were reflected in the grainy body of the control concentrates (the grain persisting despite the homogenization treatment which followed sterilization), in the color and rate of color development, and in the pronounced creaming tendency. The results are shown in Table 8. The effect of polyphosphate, particularly at the 0.6 lb per 100 lb milk solids level, in stabilizing the milk against excessive heat coagulation and subsequent excessive sediment formation, was remarkable. The grainy control concentrates were hardly of market quality to start with, and deposition of sediment both in the 26 and 36% concentrates quickly became objectionable. The concentrates containing 0.6 and 0.8 lb additive per 100 lb milk solids, even after about six months' storage at 70 degrees did not exhibit the defect to a marked degree. The sediment which formed was dispersable and would, in part, no doubt, be dispersed during commercial transport. Cream plug formation developed in all concentrates to an objectionable degree between the third and sixth month of storage, and was more pronounced in concentrates stored at 86 than in those stored at 40 F.

Concentration of additive. An optimum additive concentration of 0.6 lb per 100 lb milk solids was clearly indicated in the plant scale experiments. In a number of experiments (data not recorded) it was observed that the employment of concentrations much greater than 0.8 lb additive per 100 lb milk solids led to the production of a heavy-bodied concentrate. It is to be anticipated that variations in the positioning of polyphosphate addition in the sequence of processing steps will effect variations in results. It is also to be anticipated, in view of the results of laboratory scale experiments, that variations in the properties of the condensed phosphates, their chain length, their metal complexing capacity, and their structure (whether cyclic or linear) will bring about variations in results (2).

Heat stability. Problems engendered by heat instability become acute when concentrates are sterilized under conditions of turbulent flow, for then shear-induced effects may contribute even more than thermal effects to the coagulation process. Thus, the observation is not surprising that 26% concentrates, quite stable when sterilized in situ (data not included), show marked instability when sterilized under conditions of turbulent flow.

It may be said of the concentration-sterilization sequence of processing steps that the thermal environment sufficient for spore destruction is no less a sufficient condition for thermal coagulation in whole milk 3:1 concentrates. Thus, optimum forewarming, the use of stabilizing substances, and the employment of a high-pressure homogenization step after sterilization are indispensable if, in general, HTST processing according to a concentration followed by sterilization schedule is to be carried out successfully.

The polyphosphates are unique in the sense that they not only exercise the dual function previously mentioned, but also possess the remarkable property of peptizing insoluble protein precipitates (data not included). Thus, intractable precipitates such as one encounters in the preparation and storage of sterile and frozen milk concentrates yield to the action of polyphosphates, and more so the higher the polyphosphate concentration and the temperature.

Color. Webb and Holm (8) have observed an induction phase in the curve relating color of evaporated milk and sterilization time. In freshly prepared HTST concentrates, color development corresponds to a position on the induction phase of the curve (relating color development and time) which is determined by the thermal effects undergone by the product. Thus, concentrates which have undergone a relatively drastic heat treatment, such as the concentrates of Table 8, develop pigment at a faster rate than those which have experienced a comparatively milder treatment. No significant effect of polyphosphate on color development was observed. The color developing during storage did not match the color standards precisely (the standards were developed for conventional evaporated milk), but seemed to possess a richer yellow to green appearance.

Flavor. Panel evaluation of flavor revealed no significant differences between control and additive-containing concentrates, even when the additive concentration was as high as 1.6 lb per 100 lb of milk solids. The flavor of HTST-sterilized concentrates, in contradistinction to that of conventional evaporated milk, lends itself to improvement at the consumer level by means of suitable halogens and related oxi-

TABLE 8

Influence of polyphosphates on properties of 2:1 and 3:1 HTST sterilized milk concentrates prepared under commercial conditions. Concentrates processed according to the F-C-A-S-H sequence

Milk solids concentration	Additive[a] concentration	Properties of fresh sterile conc[b]		Properties after 55 days at:				Properties after 170 days at:			
				70 F		86 F		70 F		86 F	
		Viscosity at 30 C	Sediment	Viscosity at 30 C	Sediment	Viscosity at 30 C	Sediment	Viscosity at 30 C	Sediment	Viscosity at 30 C	Sediment
(per cent)	*(lb per 100 lb solids)*	*(centipoises)*	*(per cent)*	*(centipoises)*	[*%(inches)*]	*(centipoises)*	[*%(inches)*]	*(centipoises)*	[*%(inches)*]	*(centipoises)*	[*%(inches)*]
26	0	9.4	0	9.2	25.0(0.8)	7.3	25.0(0.8)	....[c]	50.0(1.6)	....[c]	....
26	0.4	5.2	0	4.7	1.0(0.03)	4.6	1.0(0.04)	4.3	2.2(0.07)	4.6	7.2(0.23)
26	0.6	4.9	0	4.7	0	4.6	1.5(0.05)	4.3	2.1(0.06)	4.6	6.6(0.21)
26	0.8	5.0	0	4.9	0	4.7	1.9(0.06)	4.4	3.3(0.09)	4.7	6.8(0.22)
36	0	29.4	0	50.0	48.0(1.5)	42.0	52.0(1.7)	....[c]	70.0(2.3)	....[c]	....
36	0.4	21.8	0	22.6	3.0(0.1)	20.0	5.0(0.16)	22.4	10.5(0.35)	22.8	19.9(0.61)
36	0.6	21.7	0	21.0	1.5(0.05)	19.2	2.5(0.08)	19.9	5.5(0.17)	18.1	9.0(0.29)
36	0.8	23.3	0	22.7	2.5(0.08)	22.0	4.0(0.13)	22.8	6.0(0.19)	20.3	10.8(0.35)

[a] Additive, a polyphosphate glass with an average of 4.8 phosphorus atoms per chain.

[b] Received cold 11 days after processing. Concentrate containing 26% solids and no additive—grainy; concentrate containing 36% solids and no additive—very grainy. Additive-containing concentrates were not grainy. Color index of 26 and 36% concentrates was equal to two.

[c] Too heterogeneous for measurement.

dizing additives. Iodine has been used repeatedly in our work to raise significantly the flavor level of aged HTST milks.

Storage temperature. Thermal coefficients of the rate constants governing color development, sediment formation, flavor development, thickening, and gelation appear to be of such a character that rather marked increases in stability are observed when the temperature is lowered from 86 to 70 F. Thus, the practice of storing at or near temperatures of 70 F and lower has much to commend it.

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